

(19)



Europäisches Patentamt  
European Patent Office  
Office européen des brevets

(11) Publication number:

**0 116 422**  
**A1**

(12)

## EUROPEAN PATENT APPLICATION

(21) Application number: 84300440.9

(22) Date of filing: 25.01.84

(51) Int. Cl.<sup>3</sup>: **A 61 K 7/06**  
**A 61 K 7/08, C 11 D 3/06**  
**C 11 D 17/00**

(30) Priority: 03.02.83 GB 8303014

(43) Date of publication of application:  
22.08.84 Bulletin 84/34

(84) Designated Contracting States:  
AT BE CH DE FR IT LI LU NL SE

(71) Applicant: RECKITT AND COLMAN PRODUCTS LIMITED  
P.O. Box 26 1-17, Burlington Lane  
London W4 2RW(GB)

(72) Inventor: Harmer, Jane  
2 Jessop Avenue  
Almondbury Huddersfield(GB)

(72) Inventor: Cook, Robert Victor  
11 Greenleas  
Aston on Trent Derbyshire(GB)

(74) Representative: Allard, Susan Joyce et al,  
BOULT, WADE & TENNANT 27 Fumival street  
London EC4A 1PQ(GB)

(64) Liquid cleansing compositions.

(57) Hair or body shampoo compositions having an upper aqueous layer and a lower aqueous layer which are temporarily dispersible one in the other upon agitation of the composition and wherein each layer is miscible with water in substantially all proportions. The upper layer has dissolved therein at least one detergent. The lower layer contains sodium hexametaphosphate dissolved therein. The compositions have cleansing properties and impart a conditioning effect.

EP 0 116 422 A1

BEST AVAILABLE COPY

Liquid cleansing compositions

This invention relates to multilayer liquid cleansing compositions in which one liquid phase is dispersible in another liquid phase upon shaking the composition and which on standing separate out into distinct layers. More particularly the invention relates to shampoo compositions for use on the body and hair.

Multilayer cleansing compositions have been proposed in which one layer is miscible in all proportions with water whilst another layer being of an oily nature is immiscible with water. The oily layer may comprise paraffin oil, certain esters or silicone oils. Typically the lower layer comprises a water-miscible layer including water as solvent and containing dissolved detergent whilst a water-immiscible oil-rich phase floats on the aqueous phase. Examples of such compositions include bath shampoos which are added to bath water to afford body cleansing and which provide a film of oil which is retained on the skin upon emerging from the bath to condition and/or perfume the skin; and hair shampoos where the oily layer serves to condition and/or groom the hair. An Example of such a hair shampoo occurs in United States of America Specification No. 3810478 in which the upper layer relies for its integrity upon a major amount of a mineral oil. A disadvantage of such oily materials in formulations is that they can leave an unsightly ring on the bath after use and in hair shampoos for use on naturally greasy heads are not necessarily a desirable component.

United States Patent No. 4348292 described multilayer compositions for heavy duty cleaning in the form of a single-dose concentrate for addition to water to provide a cleansing solution or dispersion. The concentrate has two aqueous layers, one layer containing at least one surfactant and the other layer containing at least one detergent builder. Preferred compositions were those in which the surfactant layer contains 50-90% by weight of surfactant and the detergent builder layer contains 30-80% by weight of detergent builder. These compositions are claimed to have enhanced cleansing efficacy as compared with homogeneous compositions, however, there is no guidance given on other than single dose presentations. In stepping from a single-dose unit to a multidose unit problems can arise in ensuring a uniform fill of each phase into the container and more importantly, in the case of a shampoo composition in ensuring that the proper dose of each phase is dispensed from the container when in use.

The specification of West German Patent Application No. 1617327 discloses a hair treatment composition containing 0.05 to 2.0% of certain metal polyphosphates of formula  $M_{n+2}P_nO_{3n+1}$ , wherein  $n$  is unspecified and  $M$  represents an alkali metal ion. Such compositions are intended to be applied to hair newly washed with soapless detergent, rinsed with water and squeeze-dried and impart conditioning effects. The specification is emphatic that the treatment is conducted as a separate step and no further rinsing is to be carried out before drying,

setting e. Furthermore, the specification advises that only ineffective amounts of the generically disclosed polyphosphates may be included into a hair shampoo formulation. A disadvantage of this disclosure is the  
5 necessity for a separate "conditioner" rinse step.

During our investigations into shampoo compositions suitable for use by those persons desirous of frequent washing of their hair, compositions were developed which were two phase and which contained a variety of detergent  
10 builders. Most unexpectedly it was found that compositions containing sodium hexametaphosphate as the detergent builder exhibit conditioning effects on hair. Moreover such compositions may be used in a conventional manner, that is, simply applied to the wetted hair then  
15 worked up to a lather and rinsed out with a copious supply of clear water. Thus the feel and the manageability of hair so washed is improved.

Accordingly the present invention provides a liquid shampoo composition having an upper aqueous layer and a  
20 lower aqueous layer, which are temporarily dispersible one in the other upon agitation and wherein each layer is miscible with water in substantially all proportions, said upper layer having dissolved therein at least one detergent and said lower layer containing sodium  
25 hexametaphosphate dissolved therein.

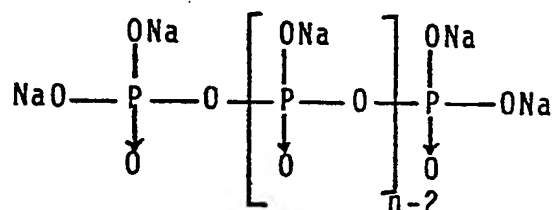
Preferably, the dispersion resulting from the agitation of the layers is stable for sufficient time to allow uniform filling of the compositions into containers so that on standing the relative ratios of the phases

within the containers are constant. This avoids the problem of having to fill each container separately with predetermined amounts of two different concentrates.

Desirably each of the upper and lower layers is substantially transparent. As will be discussed in detail later the relative ratios of the phases is dependent upon the nature and amounts of various components present.

Sodium hexametaphosphate is the term applied to a mixture of condensed phosphates of the formula:-

10



in which the average value of n is about 12, that is a selection from the generic disclosure of West German specification No. 1617327 of the case that n = 12.

In the compositions the amount of sodium hexametaphosphate will normally be at least 6% by weight of the composition.

20

Amounts of other detergent builder salts such as ammonium, sodium or potassium chlorides, sulphates, metaphosphates and/or ortho-phosphates may be included in the compositions. The total weight of the sodium hexametaphosphate plus other detergent builder salt(s) (if present) will normally comprise from 8 to 25% by weight of the composition and preferably 14-20%.

The detergent may be an anionic, cationic, amphoteric or nonionic detergent or mixtures thereof.

Preferably the compositions will include at least one anionic detergent.

The concentration of detergent in the compositions of the present invention is generally similar to those in 5 conventional shampoo compositions.

Typical of the detergents that may be used are: ammonium, substituted ammonium or sodium lauryl sulphates, or ethoxylated derivatives thereof containing up to 6 ethoxyl groups per mole; substituted ammonium such as the 10 tri- and/or diethanolamide salts of sulphated ethoxylated coconut monoethanolamide containing up to 6 ethoxyl groups per mole;  $\alpha$ -olefin sulphates and alkyl aryl sulphonates of which the high-foaming members are preferred; imidazoline betaine or cetyltrimethyl ammonium bromide. 15 The concentration of these components is conveniently in the range 8 to 25% by weight based on the total composition, and preferably in the range 10% to 18%.

In addition to the essential components mentioned above the compositions will normally include a foam 20 boosting agent such as coconut diethanolamide and optionally a superfatting agent such as ethoxylated coconut monoethanolamide. The quantities of these two components can have an effect on the relative phase volumes and the viscosity of the compositions.

25 The choice of detergent(s) in part controls the phase volume ratio of the final product. Thus, the chemical nature and the relative concentrations of the detergents employed influence the phase volume ratio. The phase volumes are also dependent upon the amount of the

sodium hexaphosphate and the amount and nature of any additional detergent builder salt. For example when the detergent is an alkyl sulphate salt and is employed in a fixed concentration of active principle in a given detergent system, a certain phase volume ratio is attained. If the anion moiety of the surfactant is ethoxylated thereby to increase the solubility thereof with the same cation, the phase volume ratio is increased that is to say that relative volume of the lower layer increases.

In the case, however, if for a particular unethoxylated anionic surfactant the nature of the cation is altered so to increase the water-solubility of the surfactant then the phase volume ratio decreases that is to say the relative volume of the lower layer decreases and in the limit it tends to disappear resulting in a homogeneous mixture. This has been found for example in the series:- sodium alkyl sulphate; ammonium alkyl sulphate; diethanolamine alkyl sulphate; triethanolamine alkyl sulphate in which series water-solubility increases from start to finish. This is a basis for finely adjusting the phase volume ratio to a predetermined value.

In order to effect an improved aesthetic appearance to the compositions one or more colouring materials may be included so that the different layers assume different colours or shades of colour to give the compositions a pleasing appearance.

The compositions may also include medicaments such as coal tar or additional conditioning agents such as

solubilised lanolin or hydrolysed proteins. The compositions may include special treatment materials which may be solids, semisolids or liquids to achieve special effects. For example an oily material or ester such as isopropyl myristate and the like may be included as a separate phase to form a multilayer product, or combined with one of the upper or lower layer phases if it is desired to produce a two layer product.

A further feature of shampoo compositions in general is that the public has grown accustomed to a degree of "thickness" consequently compositions need to have controllable viscosity of an order that may be varied to taste. A preferred range is represented by the viscosity range 1000-3000 centipoise as measured with Brookfield viscometer RVT using No. 3 spindle at speed 20 at 20°C. Clearly there may be considerable variations on both sides of this range to suit individual product requirements.

The compositions may include a proportion of an alcohol such as iso-propanol or ethanol in relatively low concentrations, of the order 2% w/w of the total composition or less, for example to solubilise a treatment compound such as coal tar, or to solubilise a perfume.

To safeguard against decomposition of components in the presence of heavy metal ions, a strong sequestering agent therefor may be incorporated without seriously disturbing the phase volume ratio. Suitable sequestering aids include ethylenediaminetetraacetic acid; the amino-methylene phosphonates such as those manufactured by Albright and Wilson under their trade mark BRIQUEST, for



example acetoethylenediaminetetramethylenephosphonic acid, and acetodiphosphonic acid; trinitriloacetic acid and the like. The free acid may be added as such or it may be added in the form of an alkali metal salt. The amount of  
 5 sequestering aid employed is of the order up to 1.00% w/w of the total composition expressed as free acid, preferably about 0.5% w/w.

The invention is illustrated by the following Examples in which percentages are weight/weight.

10 Example 1

A hair shampoo was prepared having the following composition:

	%
Triethanolamine lauryl sulphate (40% aqueous solution)	26.88
15 Ethoxylated cocomonoeethanolamide sulphate triethanolamine salt (40% aqueous solution)	8.75
Coconut diethanolamide	2.50
Ethoxylated coconut monoethanolamide	1.00
Colour, perfume	qs
20 Sodium hexametaphosphate (40% aqueous solution)	35.00
Water	balance to 100%

The composition was prepared by mixing thoroughly together with stirring all the components with the exception of coconut diethanolamide, ethoxylated cocomono-  
 25 ethanolamide and perfume which were preblended prior to addition to the rest of the components. The composition was filled directly into bottles whilst maintaining the stirring or the final filling operation can be from a well

stirred reservoir. The ratio of the volumes of the upper phase to the lower was 6:4. The viscosity of the shampoo was 1750 centipoise.

# Example 2 - 7

Shampoos of the following compositions were prepared by the method of Example 1.

	2 %	3 %	4 %	5 %	6 %	7 %
Triethanolamine lauryl sulphate (40% aqueous solution)	23.57	-	-	-	26.0	-
Ethoxylated cocomoethanolamide sulphate - triethanolamine salt (40% aqueous solution)	8.9	-	-	-	8.0	-
Coconut diethanolamide	3.0	2.5	3.0	3.0	2.5	3.5
Sodium hexametaphosphate (40% aqueous solution)	18.0	40.0	40.0	40.0	20.0	35.0
Trisodium phosphate	0.1	-	-	-	8.0	-
Sodium dihydrogenphosphate	10.0	-	-	-	-	-
Colour, perfume	qs	qs	qs	qs	qs	qs
Sodium hydroxide	qs to pH 7.0	-	-	-	-	-
Cetyltrimethylammonium bromide	-	14.0	-	-	-	-
Ethoxylated coconut monoethanolamide	-	1.0	0.75	0.75	1.0	2.0
Diethanolamine lauryl sulphate (33% aqueous solution)	-	-	42.65	-	-	-
Dodecylbenzenesulphonate - triethanolamine salt (60% aqueous solution)	-	-	-	24.17	-	-
Imidazoline betaine (40% aqueous solution)	-	-	-	-	-	36.0
Water	balance to 100					

0116422

The seven Examples afford mild shampoos with gentle cleansing agents which cleanse the hair without stripping it of its natural oil and which contain non oily conditioners to maintain its condition. They are also  
5 useful for people who wash their hair very frequently and only a single application is necessary. Thus excessive defatting of the hair and/or skin is obviated.

Examples 8, 9

Shampoos of the following compositions were prepared  
10 by the method of Example 1.

	<u>8</u>	<u>9</u>
	%w/w	%w/w
Triethanolamine lauryl sulphate	26.9	26.9
Acylaminopolyglycolether sulphate, triethanolamine salt	8.8	8.8
15 Coconut diethanolamide	2.5	2.5
Ethoxylated coconut monoethanolamide	1.0	1.0
Sodium hexametaphosphate	16.0	16.0
Acetodiphosphonic acid	0.5	-
Ethylenediaminetetracetic acid	-	0.5
20 Perfume	qs	qs
Colourant	qs	qs
Preservative	qs	qs
Water	ad 100% ad 100%	

These Examples produce mild shampoo compositions of  
25 pH about 6.5, the components of which are at acceptable concentrations and the compositions are of a viscosity to be expected by users. They also are useful for people who

" wash their hair very frequently and by a single application is necessary at each washing. Thus excessive defatting of the hair and/or skin is obviated.

CLAIMS:

1. A liquid shampoo composition having an upper aqueous layer and a lower aqueous layer, which are temporarily dispersible one in the other upon agitation and wherein each layer is miscible with water in substantially all proportions, said layer having dissolved therein 8% to 25% by weight of the composition of at least one detergent and said lower layer containing at least 6% by weight of the composition of sodium hexametaphosphate dissolved therein.
2. A shampoo composition according to claim 1 wherein a detergent builder salt selected from ammonium, sodium or potassium chlorides, sulphates, metaphosphates or orthophosphates is present in amount such that together with the sodium hexametaphosphate it represents 8% to 25% by weight of the composition.
3. A shampoo composition as claimed in claim 2 wherein the total weight of sodium hexametaphosphate and optional detergent builder salt(s) is from 14 to 20% by weight of the composition.
4. A shampoo composition as claimed in any preceding claim wherein the detergent is selected from ammonium, substituted ammonium or sodium lauryl sulphates, or ethoxylated derivatives thereof containing up to 6 ethoxyl groups per mole; substituted ammonium such as the tri- and/or diethanolamide salts of sulphated ethoxylated

coconut monoethanolamide containing up to 6 ethoxyl groups per mole;  $\alpha$ -olefin sulphates and alkyl aryl sulphonates; imidazoline betaine or cetyltrimethyl ammonium bromide.

5        5.        A shampoo composition as claimed in any one of the preceding claims wherein the total weight of the detergent(s) is from 10 to 18% by weight of the composition.

10       6.        A shampoo composition as claimed in any one of the preceding claims wherein the dispersion resulting from the agitation of the layers has a viscosity of up to 3000 centipoise when measured on a Brookfield viscometer model RVT using spindle No. 3 at speed 20 at 20°C.

15       7.        A shampoo composition as claimed in claim 6 wherein the viscosity of the said dispersion is in the range of from 1000 to 3000 centipoise.

20       8.        A shampoo composition as claimed in any one of the preceding claims wherein the dispersion resulting from the agitation of the layers has a pH of from 6 to 7.

9.        A shampoo composition as claimed in any one of the preceding claims which additionally contains a sequestering agent which is distributed between the two layers.

25       10.       A shampoo composition as claimed in claim 9 wherein the sequestering agent is ethylenediamine tetracetic acid or an aminomethylene phosphonate.

11. A method of washing hair which comprises shaking a shampoo composition as claimed in any one of the preceding claims to form a temporary dispersion, applying said so shaken composition to moistened hair, working to a lather and rinsing with clear water whereby the hair is both washed and conditioned.
12. A method for the preparation of a shampoo composition as claimed in any one of Claims 1 to 10 claims which comprises blending together at least one detergent in an amount to provide from 8% to 25% by weight of the total composition, sodium hexametaphosphate in an amount to provide at least 6% by weight of the total composition and any other desired ingredients, adding water in the amount required to bring the composition to 100% by weight, stirring the mixture of components until a uniform dispersion is obtained and filling the so-produced composition into containers.
13. A method for the preparation of a shampoo composition as claimed in any one of Claims 1 to 10 which comprises blending a 40% aqueous solution of at least one detergent and a 40% aqueous solution of sodium hexametaphosphate, optionally with the addition of any other desired ingredients, with water to form a uniform dispersion, the detergent forming 8% to 25% by weight of the total composition and the sodium hexametaphosphate forming at least 6% by weight of the total



composition, and filling the so-produced composition into containers.



DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
D,Y	US-A-4 348 292 (MARTIN E.GINN) *Claims; column 4, lines 44-49*	1	A 61 K 7/06 A 61 K 7/08 C 11 D 3/06 C 11 D 17/00
Y	FR-A- 857 207 (COLGATE-PALMOLIVE) *Example V*	1	
D,Y	US-A-3 810 478 (FRANK WESLEY OLSON et al.) *Claims*	1	
A	US-A-4 107 067 (ALAN PEARCE MURPHY et al.) *Claims 1,6*	1	
D,A	DE-A-1 617 327 (BENCKISER) *Claim 1*	1	
The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (Int. Cl. 3)
Place of search THE HAGUE		Date of completion of the search 30-03-1984	Examiner MALHERBE Y.J.M.
<b>CATEGORY OF CITED DOCUMENTS</b>			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☒ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☐ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**